

# Crystal structures of two thienyl analogs of benzil – 1,2-dithien-2-ylethanedione (2,2'-thenil) and 1,2-dithien-3-ylethanedione (3,3'-thenil)

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2,2'-Thenil crystallizes in  $P2_1/c$  with  $a = 7.2501(12)$  Å,  $b = 4.7846(8)$  Å,  $c = 13.9867(23)$  Å,  $\beta = 96.897(3)^\circ$ ,  $V = 481.67(14)$  Å<sup>3</sup>, and  $Z = 2$ . The molecule resides on an inversion center and is planar. 3,3'-Thenil also crystallizes in  $P2_1/c$  with  $a = 3.9904(8)$  Å,  $b = 21.310(4)$  Å,  $c = 11.618(2)$  Å,  $\beta = 101.83(3)^\circ$ ,  $V = 966.9(3)$  Å<sup>3</sup>, and  $Z = 4$ . Refinement of 3,3'-thenil data indicated that 10.3(2)% of both thienyl rings are flip-disordered in this nonplanar molecule. A brief discussion of disorder in molecules containing terminal, unsubstituted 2- and 3-thienyl rings is presented.

**KEY WORDS:** Thenil; benzil; molecular structure; disorder.

## Introduction

At room temperature, pure benzil crystallizes in  $P3_12$  (or  $P3_22$ ) and appears uniaxial when viewed normal to (001) faces.<sup>1</sup> However in the late 1800s, the French mineralogist Wyruboff reported that hexagonal crystals of benzil, when viewed down the  $c$ -axis, were biaxial and divided into six distinct and symmetrically related sectors upon inspection between crossed polars in a polarizing microscope.<sup>2</sup> During the turn of the 19th century, many other optically anomalous organic crystals were reported by optical mineralogists.<sup>3</sup> Nearly a century later, some of these optically anomalous, sectored, organic crystals were duplicated by adding isomorphous impurities to puri-

fied hosts; however, the cause of the anomalous birefringence that caused the stark sectoring in benzil has never been determined.<sup>3</sup> Researchers have used minor amounts of benzoin and deoxybenzoin as impurities during benzil crystallization but were unable to recreate Wyruboff's curious observation.<sup>3</sup>

Since thiophene has a similar molecular volume as benzene, guest molecules containing thienyl rings could be used to examine host-guest interactions during crystal growth since guests can alter the morphology of host crystals and/or incorporate into the bulk of the crystal.<sup>4</sup> When forming mixed crystals, thienyl-containing guest have incorporated into the bulk by keeping non-aromatic based lone pairs on the thienyl-ring sulfurs pointed away from the face of the growing crystal presumably to avoid unfavorable electrostatic interactions.<sup>5</sup> Research in our lab has centered on syntheses and crystal structure of thienyl-analogs of phenyl-containing molecules commonly encountered in

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**Table 1.** Crystal Data and Structure Refinement Information for 2,2'- and 3,3'-Thenils

Compound	2,2'-Thenil	3,3'-Thenil
IUPAC name	1,2-dithien-2-ylethanedione	1,2-dithien-3-ylethanedione
CCDC no.	182707	182706
Color/shape	Orange–yellow needle	Yellow needle
Chemical Formula	C <sub>10</sub> H <sub>6</sub> O <sub>2</sub> S <sub>2</sub>	C <sub>10</sub> H <sub>6</sub> O <sub>2</sub> S <sub>2</sub>
Formula weight	222.27 g/mol	222.27 g/mol
Melting point	81°C	76°C
Temperature	294(2) K	293(2) K
Crystal system	Monoclinic	Monoclinic
Space group	<i>P</i> 2 <sub>1</sub> / <i>c</i>	<i>P</i> 2 <sub>1</sub> / <i>c</i>
Unit cell dimensions	<i>a</i> = 7.2501(12) Å <i>b</i> = 4.7846(8) Å <i>c</i> = 13.9867(23) Å $\beta$ = 96.897(3)°	<i>a</i> = 3.9904(8) Å <i>b</i> = 21.310(4) Å <i>c</i> = 11.618(2) Å $\beta$ = 101.83(3)°
Volume	481.67(14) Å <sup>3</sup>	966.9(3) Å <sup>3</sup>
Z	2	4
Density (calculated)	1.533 g/cm <sup>3</sup>	1.527 g/cm <sup>3</sup>
Absorption coefficient	0.518 mm <sup>-1</sup>	0.516 mm <sup>-1</sup>
Extinction coefficient	0.006(10)	0.0050(16)
Diffractometer/scan	Siemens SMART/CCD	Siemens SMART/CCD
$\theta$ range for data collection	2.83–24.71	1.91–28.27
Reflections measured	2206	10084
Independent/observed reflections	810 ( <i>R</i> <sub>int</sub> = 0.032/574 [ <i>I</i> > 2σ( <i>I</i> )])	2294 ( <i>R</i> <sub>int</sub> = 0.067/1303 [ <i>I</i> > 2σ( <i>I</i> )])
Data/restraints/parameters	810/0/65	2294/23/142
Goodness of fit on <i>F</i> <sup>2</sup>	1.015	1.030
Final <i>R</i> indices [ <i>I</i> > 2σ( <i>I</i> )]	<i>R</i> 1 = 0.0520, <i>wR</i> 2 = 0.1316	<i>R</i> 1 = 0.0580, <i>wR</i> 2 = 0.1002
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0784, <i>wR</i> 2 = 0.1467	<i>R</i> 1 = 0.1292, <i>wR</i> 2 = 0.1179

undergraduate organic chemistry labs with the ultimate goals of investigating organic crystal growth. To date, we have published crystal structures of both disubstituted thienyl analogs of benzoin—1,2-dithien-2-ylhydroxyethanone and 1,2-dithein-3-ylhydroxyethanone, as well as crystal structures of 2,3-dithien-2-ylquinoxaline and 1,4-diphenyl-1-2,3-dithein-3-ylcyclopentadien-1-one.<sup>6</sup> This paper describes the crystal structures of two dithienyl analogs of benzil—1,2-dithien-2-ylethanedione (2,2'-thenil) and 1,2-dithien-3-ylethanedione (3,3'-thenil).<sup>7(3)</sup> Since thiophenes were common impurities in benzene distillates during Wyruboff's times, it seems altogether likely that thienyl-impurities could have caused the anomalous sectoring in benzil.

<sup>(3)</sup>Early researchers (see references 7) on Dithienyl-ethanediones came up with the “thenil” moniker through their interpretation of IUPAC rule C313.4.

## Experimental section

The abovementioned disubstituted thenils were made in adequate quantities through the base-assisted condensation of corresponding thiophenecarboxaldehydes in ethanol using thiamine as a catalyst followed by oxidation using copper(II) acetate in acetic acid.<sup>8</sup> Recrystallization from 95% ethanol yielded orange–yellow needles of 2,2'-thenil, mp 81°C.<sup>7a–c</sup> [Additional data for 2,2'-thenil – IR(Fluoromac, cm<sup>-1</sup>): 2950 (m), 2850 (m); IR(Nujol, cm<sup>-1</sup>): 1700 (s), 1450 (m), 1380 (s); <sup>1</sup>H NMR(CDCl<sub>3</sub>, ppm): 8.05 (d, 1H), 7.82 (d, 1H), 7.19 (t, 1H)] For 3,3'-thenil, recrystallization from 95% ethanol yielded golden yellow needles with a melting point 80°C.<sup>7</sup> [Additional data for 3,3'-thenil— IR(Fluoromac, cm<sup>-1</sup>): 2950 (m), 2850 (m); IR(Nujol, cm<sup>-1</sup>): 1700 (s), 1450 (m), 1380 (s); <sup>1</sup>H NMR(CDCl<sub>3</sub>, ppm): 8.33 (d, 1H), 7.67 (d, 1H), 7.37 (m, 1H)] Samples for crystal structure determination were excised

**Table 2.** Table of Fractional Coordinates and Equivalent Isotropic Thermal Parameters for 2,2'- and 3,3'-Thenil

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>iso</sub>
2,2'-Thenil				
C1	0.1034(5)	0.4639(8)	0.0006(3)	0.0552(10)
O1	0.1919(3)	0.5785(5)	−0.0567(2)	0.0722(9)
S1	0.07627(13)	0.0927(2)	0.15733(7)	0.0649(5)
C2	0.1866(4)	0.2617(6)	0.0721(2)	0.0498(9)
C3	0.3669(5)	0.1835(7)	0.0759(3)	0.0533(9)
C4	0.4156(5)	−0.0143(8)	0.1499(3)	0.0642(11)
C5	0.2724(6)	−0.0798(8)	0.1992(3)	0.0671(11)
3,3'-Thenil				
C1	0.1125(7)	0.34753(14)	1.2627(2)	0.0414(7)
O1	0.0314(6)	0.29522(10)	1.28833(17)	0.0632(7)
C6	0.1367(8)	0.39903(13)	1.3558(2)	0.0400(7)
O2	−0.0241(6)	0.44738(10)	1.32812(17)	0.0602(7)
S1A	0.3137(5)	0.42263(5)	0.97101(9)	0.0527(3)
S1B	0.236(7)	0.3491(6)	0.9292(9)	0.0527(3)
C2A/C4B	0.2589(8)	0.42207(13)	1.1116(2)	0.0442(8)
C3A/C3B	0.1719(7)	0.36384(12)	1.1457(2)	0.0368(7)
C4A/C2B	0.1508(8)	0.31914(14)	1.0546(2)	0.0472(8)
C5A	0.217(2)	0.3441(2)	0.9571(4)	0.0528(11)
C5B	0.287(18)	0.4203(9)	0.9973(19)	0.0528(11)
S2A	0.5795(3)	0.40297(5)	1.69199(7)	0.0538(4)
S2B	0.694(6)	0.3350(9)	1.6596(7)	0.0538(4)
C7A/C9B	0.3506(7)	0.42959(13)	1.5632(2)	0.0435(7)
C8A/C8B	0.3336(7)	0.38685(12)	1.4744(2)	0.0341(6)
C9A/C7B	0.5200(7)	0.33163(13)	1.5139(2)	0.0437(7)
C10A	0.6685(18)	0.3337(2)	1.6298(3)	0.0420(10)
C10B	0.581(9)	0.4121(10)	1.660(2)	0.0420(10)

from single crystals displaying uniform birefringence. All spectroscopies were performed at room temperature.

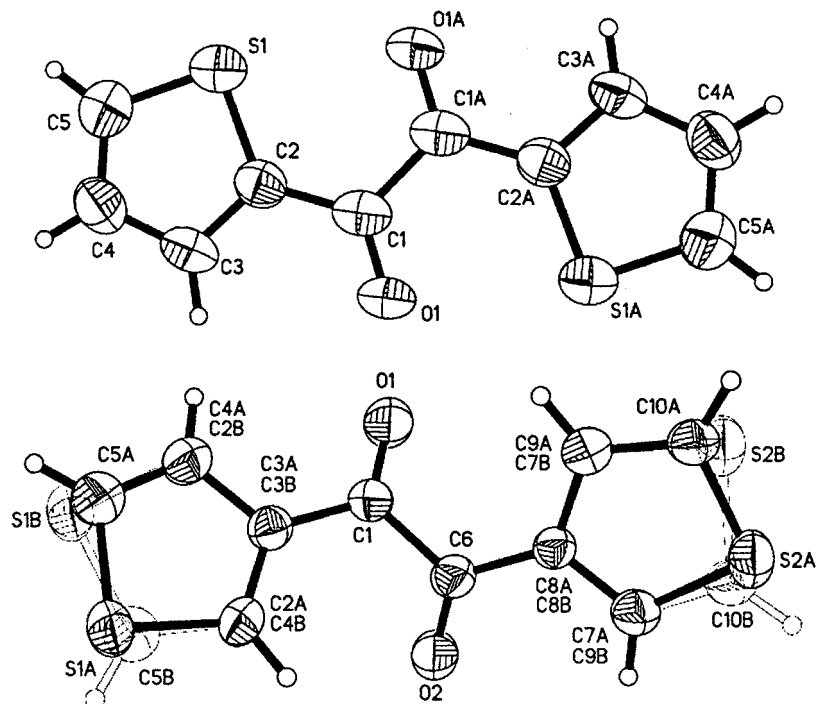
Single-crystal data were collected at room temperature using a Siemens (Bruker) 3-Circle diffractometer equipped with a CCD detector utilizing the SMART software package.<sup>9</sup> The data was integrated using SAINT.<sup>10</sup> No absorption corrections were applied to data.<sup>(4)</sup> Structure refinement and solution was performed using SHELXTL.<sup>11</sup> Hydrogen atoms were generated at fixed distances of 0.9300 Å for thienyl-ring carbons. Additional refinement details are given under Results.

## Results

Refinement details for both thenils are shown in Table 1 and atomic coordinates and isotropic thermal parameters are listed in Table 2. Inspection of bond lengths and angles during refinement suggested that 3,3'-thenil displayed a thienyl-ring flip disorder. Initial models indicated that the percent disorder was similar for each ring; therefore, a single occupancy variable was refined and revealed 10.3(2)% occupancy for the flipped position. (The disordered rings are shown in gray in Fig. 1.) To model the disorder and retain ideal thiophene ring bond lengths and angles, the authors took the following steps:<sup>6b,12</sup>

- 1) The flip-disordered sulfurs (S1B and S2B) were identified from difference maps. These atoms were very close to thienyl-ring carbons C5A and C10A.

<sup>(4)</sup>The authors did not apply absorption corrections since the samples were adequately bathed in the X-ray beam. (Crystal size in all dimensions was less than the 5-mm X-ray beam collimator.)



**Fig. 1.** The ORTEP drawings of 2,2'-thienil (top) and 3,3'-thienil (bottom) shown with 50% probability ellipsoids. The disorder thienyl rings in 3,3'-thienil are shown in gray.

- 2) Atoms in each ring were duplicated and designated as the appropriate flipped carbon atom (*e.g.* for the first ring, S1A, C2A, C3A, and C4A became C5B, C4B, C3B, and C2B respectively).
- 3) Atomic coordinates and thermal parameters were fixed for the three sets of ring carbon atoms (*e.g.* C2A/C4B, C3A/C3B, and C4A/C2B) that remained stationary in each flipped component.
- 4) Though spatially different, the thermal parameters for the remaining carbon and sulfur atoms in the rings were set to be equal (*e.g.* S1A/S1B and C5A/C5B).
- 5) The bond distances between sulfur atoms and neighboring carbons were constrained to ideal S—C thienyl-ring distances of 1.701 Å and the bond distances between C4A—C5A, C4B—C5B, C9A—C10A, and C9B—C10B were constrained to typical bond lengths of 1.345 Å.

Bond lengths for both molecules fall within expected ranges and are shown along with angles in Table 3. 3,3'-Thienil is not planar because of steric effects and is therefore more analogous to benzil. For 3,3'-thienil, the torsion angle (O1—C1—C6—O2) is 126.9(6)° whereas the many crystal structure determinations of benzil have given similar torsion angles that average 108.1° (1.8).<sup>1</sup> In the crystal structure of 2,2'-thienil, the molecule resides on an inversion center and yields a completely planar molecule with a O1—C1—C1A—O1A torsion angle of 180.0°. This result is not surprising considering the lack of steric hindrance between thienyl-ring hydrogens and neighboring carbonyl groups. A search of the April 2002 release of the Cambridge Structural Database (CSD) for other di-substituted aromatic ethanediones presented the structures of 32 molecules whose ethanedione fragment was itself not part of a ring system.<sup>13</sup> Five entries of the 32 had aromatic substituents that sterically prevented the attached aromatic ring from

**Table 3.** Table of Selected Geometric Relationships for 2,2'- and 3,3'-Thenil

		2,2'-thenil	3,3'-thenil	
Bonds	C1—O1	1.216(4) Å	C1—O1	1.215(3) Å
	C1—C1A	1.537(7) Å	C6—O2	1.222(3) Å
	C1—C2	1.468(5) Å	C1—C6	1.530(4) Å
	S1—C2	1.715(3) Å	C1—C3A	1.469(4) Å
	S1—C5	1.687(4) Å	C6—C8A	1.462(4) Å
	C2—C3	1.354(4) Å	S1A—C2A	1.692(3) Å
	C3—C4	1.416(5) Å	S1A—C5A	1.718(4) Å
	C4—C5	1.351(5) Å	C2A—C3A	1.369(4) Å
Angles			C3A—C4A	1.413(4) Å
			C4A—C5A	1.327(4) Å
			S2A—C7A	1.683(3) Å
			S2A—C10A	1.713(4) Å
			C7A—C8A	1.368(4) Å
			C8A—C9A	1.417(4) Å
			C9A—C10A	1.355(3) Å
	O1—C1—C1A	118.8(4)°	O1—C1—C6	117.8(3)°
	O1—C1—C2	122.7(3)°	O1—C1—C3A	123.1(3)°
	C2—S1—C5	91.60(18)°	O2—C6—C1	117.7(2)°
	S1—C2—C3	111.8(3)°	O2—C6—C8A	123.3(3)°
	C2—C3—C4	111.6(3)°	C2A—S1A—C5A	90.76(17)°
	C3—C4—C5	112.8(3)°	S1A—C2A—C3A	112.1(2)°
	C4—C5—S1	112.2(3)°	C2A—C3A—C4A	112.0(2)°
			C3A—C4A—C5A	112.2(3)°
			C4A—C5A—S1A	112.9(3)°
Torsion angles	O1—C1—C1A—O1A	180.0°	C7A—S2A—C10A	92.13(17)°
			S2A—C7A—C8A	112.3(2)°
			C7A—C8A—C9A	111.5(2)°
			C8A—C9A—C10A	112.9(3)°
			C9A—C10A—S2A	111.1(3)°
			O1—C1—C6—O2	126.9(6)°

being planar with its nearest carbonyl group, whereas the other 27 had aromatic rings that were coplanar with the nearest carbonyl group.<sup>(5)</sup> From this set of 27, only one entry had aromatic rings *and* carbonyl moieties in a completely planar environment like 2,2'-thenil [rac-5,5'-bis(2-

acetoxypentyl)-2,2'-furyl, CSD Reference Code: COCJUZ].<sup>14</sup> The remaining entries, including 2,2'-furyl, were all nonplanar molecules, with ethanedione torsion angles ranging from 94.19° to 130.99°.

#### *Disorder in Thienyl Rings—A Study of the Cambridge Structural Database(CSD)*

Recently, researchers have commented on the probability of flip disorder in molecules having at least one unsubstituted 2-thienyl moiety. Their search on an unspecified version of the CSD yielded 130 hits on the word “disorder” out of 378 molecules with terminal unsubstituted 2-thienyl rings.<sup>15</sup> However, the authors did not determine whether all disorder flags were due to

<sup>(5)</sup>Ethanediones were chosen if (1) they had aromatic rings on both ends of the molecule, (2) they only had a single ethanedione center, and (3) they did not have a ethanedione moiety that was itself a part of a ring system. The 32 entries for di-substituted aromatic ethanediones are listed alphabetically as follows by CSD Reference Code: BENZIL, BENZIL01, BENZIL02, BENZIL03, BENZIL04, BENZIL05, BOLCUA\*, CASGEI, CASGEI01, CASGIM, CASGIM1, CASGOS, CASGUY, CASHAF, COCJUZ, DACJAS, DNBZIL, FIYVUE, GEBNAC, HXBZLA\*, JAVLEX, JAVLUN, LATWOS, MEKDOV, PPYRED, PYRDIL, PYRDIL01, SIWDEH\*, SIWDEH01\*, WETVIA, WOTMUN, ZIXCAK\*. (Asterisked entries have planar ethanedione moieties with orthogonal aromatic rings.)

flipped rings. The authors state that a telling sign of 2-thienyl ring-flip disorder is a bond angle at the ring carbon atom opposite of the sulfur that deviates noticeably from  $112^\circ$ .<sup>15</sup> [In 2,2'-thienyl the corresponding bond angle (C2—C3—C4) is  $111.6(3)^\circ$ .] In addition to anomalous angles, we have found that elongated aromatic carbon-carbon bond distances are prevalent in molecules with disordered 3-thienyl rings.<sup>6b</sup> Therefore, we have expanded the search for molecules exhibiting 2- and 3-thienyl flip disorders in the April 2002 release of the CSD. A total of 682 total hits were obtained during a search for molecules containing at least one terminal, unsubstituted thienyl ring. [Of the total hits, 628 correspond to molecules containing at least one 2-thienyl pendant, 50 to molecules with a 3-thienyl ring, and 4 molecules with both types of rings]. Of these published molecules, 252 have specific mention of disorder with thienyl rings [233 for 2-thienyl, 18 for 3-thienyl, and 1 for molecules with both rings]. Overall, 37% of total entries mention the treatment of thienyl-ring flip disorder during refinement. As previous researchers have stated in their initial CSD search on 2-thienyl ring systems, there is probably a significant number of structures containing disorder that was not addressed during refinement.<sup>14(6)</sup>

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<sup>(6)</sup> During the sorting of entries in our search for disorder, one example immediately became apparent. The structure of dichloro-(*N,N'*-bis (1-methylene-thiophene)-1,3-diaminopropane)-palladium(II) dichloromethane solvate [CSD Reference Code: YAZMAN] mentions no disorder yet has one 2-thienyl ring with carbon-carbon bond lengths of 1.372 Å, 1.319 Å, and 1.577 Å. The same ring has a  $102.6^\circ$  angle at the ring carbon atom opposite of the sulfur.

## References

- (a) Brown, C.J.; Sadanaga, R. *Acta Cryst.* **1965**, *18*, 158–164. (b) Gabe, E.J.; Le Page, Y.; Lee, F.; Barclay, L.R.C. *Acta Crystallogr., Sect. B*, **1981**, *37*, 197–200. (c) More, M.; Odou, G.; Lefebvre, J. *Acta Cryst., Sect. B*, **1987**, *43*, 398–405.
- Wyruboff, M.G. *Bull. Soc. Min. Fr.* **1884**, *7*, 84–88.
- McBride, J.M.; Kahr, B. *Angew. Chim. Int. Ed. Eng.*, **1992**, *31*, 1–110 and references therein.
- Vaida, M.; Shimon, L.J.W.; Weisinger-Lewin, Y.; Frolow, F.; Lahav, M.; Leiserowitz, L.; McMullan, R.K.; *Science*, **1988**, *241*, 1475–1479.
- Shimon, L.J.W.; Vaida, M.; Frolow, F.; Lahav, M.; Leiserowitz, L.; Weisinger-Lewin, Y.; McMullan, R.K. *Faraday Discuss.*, **1993**, *95*, 307–327.
- (a) [1,2-Di-2-thienyl-2-hydroxyethanone] Crundwell, G.; Meskill, T.; Sayers, D.; Kantardjieff, K. *Acta Cryst.* **2002**, *E58*, o666–o667. (b) [1,2-Di-3-thienyl-2-hydroxyethanone] Crundwell, G.; Meskill, T.; Sayers, D.; Kantardjieff, K. *Acta Cryst.* **2002**, *E58*, o668–o670. (c) [2,3-dithien-2-ylquinoyaline] Crundwell, G.; Sayer, D.; Herron, S.R.; Kantardjieff, K. *Acta Crystal.* **2003**, *E59*, 0314–0315. (d) [1,4-diphenyl-2,3-dithien-3-ylcyclopentadien-1-one] Linehan, J.; Crundwell, G.; Herron, S.R.; Kantardjieff, K. *Acta Cryst.* **2003**, *E59*, 0466–0468.
- [2,2'-thienyl] (a) Marchese, G.; Babudri, F.; Fiandanese, V.; Punzi, A. *Tetrahedron Lett.*, **1995**, *36*, 7305–7308. (b) Schuetz, R.D.; Nilles, G.P. *J. Org. Chem.* **1971**, *36*, 2486–2489. (c) Cardon, S.Z.; Lankelma, H.P.; *J. Am. Chem. Soc.* **1948**, *70*, 4248–4249. [3,3'-thienyl] (d) Campagne, E.; Bourgeois, R.C. *J. Am. Chem. Soc.* **1953**, *75*, 2702–2703.
- Although many texts have these classic experiments, the following two texts were used as references. [Aldehyde condensation] (a) Pavia, D.L.; Lampman, G.M.; Kris, G.S.; Engel, R.G. *Organic Laboratory Techniques: A Microscale Approach*, 2nd Ed.; Saunders College Publishing: New York, 1995; pp 282–285. [Oxidation to ethanedione] (b) Lehman, J.W. *Operational Organic Chemistry: A Laboratory Course*, 2nd Ed.; Prentice Hall: New York, 1988, pp 500–501. More direct and elegant reactions are found in the literature with higher yields (see references 2a–b); however, the authors felt these above pathways were suitable for undergraduates starting independent research.
- SMART: Siemens Energy & Automation, Inc. (Bruker); Madison, WI, 1996.
- SAINT Version 4.0 Software Reference Manual: Siemens Energy & Automation, Inc. (Bruker); Madison, WI, 1996.
- Sheldrick, G. M. SHELXL97, *Program for Crystal Structure Refinement*; University of Göttingen, Germany, 1997. Sheldrick, G.M. *Acta Crystallogr. Sect. A*, **1990**, *A46*, 467–473.
- (a) Antolini, L.; Tedesco, E.; Barbarella, G.; Favaretto, L.; Sotgiu, G.; Zambianchi, M.; Casarini, D.; Gigli, G.; Cingolani, R. *J. Am. Chem. Soc.* **2000**, *122*, 9006–9013. (b) Barbarella, G.; Zambianchi, M.; Antolini, L.; Ostojka, P.; Maccagnani, P.; Bongini, A.; Marseglia, E.A.; Tedesco, E.; Gigli, G.; Cingolani, R. *J. Am. Chem. Soc.* **1999**, *121*, 8920–8926.
- Allen, F.H.; Kennard, O. *Chem. Design Automat. News* **1993**, *8*, 1, 31–37.
- Handrosch, C.; Heinemann, F.W.; Kisch, H. *Acta Cryst., Sect. C* **1999**, *55*, 2184–2186.
- Zheng, Z.; Wang, J.; Liu, H.; Carducci, M.D.; Peyghambarian, N.; Jabbour, G.E. *Acta Cryst., Sect. C* **2002**, *58*, m50–m52.